Performance Monitoring Protocol (QA/QC) for the Agilent GC/MS

1 Scope

This document addresses the performance monitoring (QA/QC) of the Agilent GC/MS system which may include optional detectors, such as a Flame Ionization Detector (FID). This document applies to personnel using the associated instrument(s)/equipment in Quantico, VA in the following disciplines/categories of testing: Drug chemistry, toxicology, paint, explosives (chemistry), fire debris, and Chemistry Unit general physical and chemical analysis.

2 Principle

The Agilent GC/MS system consists of an Agilent Gas Chromatograph (GC) with a single quadrupole Mass Selective Detector (MSD) Mass Spectrometer (MS). The system may also be equipped with an additional detector, such as an FID. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

The mass spectrometer will be configured to perform specific modes of ionization depending on which of the two types of ion sources is installed. If the electron impact (EI) ionization source is installed, only positive ion EI ionization analysis will be performed. However, if the chemical impact (CI) ionization source is installed, then either positive ion CI (PICI) or negative ion CI (NICI) analyses may be performed.

3 Equipment/Materials/Reagents

a. Instrumentation - Agilent 7890 GC, 5975 or 5977 MSD with EI or CI Source, FID (if equipped), and MSD Chemstation software (or equivalent)

b. Autosampler - Agilent ALS, CTC "Pal" Series, or Gerstel MPS automated sampler, accessories, and software (or equivalent)

c. GC Column (MSD) – Agilent J&W DB-5 MS, 30 m, 0.25 mm i.d., 0.25 µm film (or equivalent)

d. GC Column (FID) – Agilent J&W DB-5, 15 m, 0.25 mm i.d., 0.25 µm film (or equivalent)

e. Carrier Gas - Helium, 99.99% (high purity)

f. CI Reagent Gas - Methane, 99.99% (high purity)

g. Compressed air

h. Hydrogen Gas, 99.99% (high purity)

i. Nitrogen Gas, 99.99% (high purity)

j. Chloroform, GC grade

k. Lidocaine HCl (Sigma or equivalent)

l. Tributoxyethyl Phosphate (TBEP) (Chem Service or equivalent)

m. Perfluorotributylamine (PFTBA, FC-43) (Agilent or equivalent)

n. Perfluoro-5,8-dimethyl-3,6,9-trioxidodecane (PFDTD) Tuning Solution (Agilent or equivalent)

o. Analytical balance

p. Volumetric flask

q. Autosampler vials - 2 mL GC vials, crimp or screw top, with or without 100-500 µL inserts (Agilent or equivalent)

r. Injection port liners - 4 mm split-splitless, tapered, with or without glass wool (Agilent or equivalent)

s. Injection port septa - standard low-bleed 11 mm (Agilent or equivalent)

t. Autosampler syringes - Hamilton 701ASN 10 µL (or equivalent)

4 Standards and Controls

4.1 PFTBA Tuning Solution

The PFTBA tuning solution is used for tuning the mass spectrometer and verifying mass assignment and accuracy when the EI source is installed. It is supplied by the instrument manufacturer and does not expire. It is stored in a glass container attached to the MSD. Under normal conditions, this should not need to be refilled.

4.2 PFDTD Tuning Solution

The PFDTD tuning solution is used for tuning the mass spectrometer and verifying mass assignment and accuracy when the CI source is installed. It is supplied by the instrument manufacturer and does not expire. It is stored in a glass container attached to the MSD. Under normal conditions, this should not need to be refilled.

4.3 Testmix (0.05 mg/mL each of Lidocaine and TBEP)

The testmix is used to assess daily operating performance, mass assignment, and continued integrity of the system. To prepare, weigh 5.8 mg Lidocaine HCl and 5 mg TBEP into a 100-mL volumetric flask. Bring to the mark with chloroform and mix well. Store the solution in the refrigerator. It has a shelf-life of three years. This preparation may be appropriately scaled up.

5 Sampling or Sample Selection

Not applicable.

6 Procedures

6.1 Daily Checks

The following steps will be performed daily, regardless of the ion source installed, mode of ionization, or the detector to be used. Enter the appropriate information in the QA/QC log for tracking purposes.

a. Check to ensure that the GC wash vials are filled, the waste vials are empty, and all are in the appropriate positions.

b. Record the remaining disk space on the hard drive. Use the Windows Explorer program to verify that the hard disk has at least 100 MB of free disk space. Do not use if less than 100 MB remain.

c. Record the line pressure of the building helium supply (carrier gas). The regulator should read 50 p.s.i. or above. If it cannot maintain this pressure, contact appropriate instrument support personnel. If the helium is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100 p.s.i. remaining.

6.1.1 EI Source Daily Checks

If using the MSD with the EI source installed, perform the following steps:

a. Check the Ion Gauge to ensure that there are no significant leaks in the system. Do not use if the source pressure is higher than 6×10^{-5} torr.

b. Perform a tune of the instrument. If Autotune (ATUNE) is selected, the mass spectrometer will tune itself using PFTBA. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, save and print the tune file (ATUNE) when completed.

c. Perform an analysis of the testmix. Open the appropriate testmix instrument method, and verify the parameters as listed in the 'Instrumental Conditions' section of this protocol. Set up a sequence, load the autosampler with a vial containing the testmix, and start the analysis. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the TIC and mass spectra for both TBEP and Lidocaine.

d. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact appropriate instrument support personnel.

6.1.2 CI Source Daily Checks

If using the MSD with the CI source installed, perform the following steps:

a. Record the tank pressure of the methane tank (reagent gas). Change the tank if less than 100 p.s.i. remaining.

b. Check the Ion Gauge to ensure that there are no significant leaks in the system. Do not use if the source pressure is higher than the following:

- PICI analysis - 6×10^{-4} torr with reagent gas on at approximately 20%
- NICI analysis – 6×10^{-4} torr with reagent gas on at approximately 40%

c. Perform an analysis of the testmix. Open the appropriate testmix instrument method, and verify the parameters as listed in the 'Instrumental Conditions' section of this protocol. Set up a sequence, load the autosampler with a vial containing the testmix, and start the analysis. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the TIC and mass spectra for both TBEP and Lidocaine.

d. If sample analyses will be performed using negative ion mode, no additional daily checks will be required.

e. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact appropriate instrument support personnel.

6.1.3 FID Daily Checks

If using the FID, perform the following steps:

a. Record the line pressure of the hydrogen supply from the generator. The value should read 50 p.s.i. or above. If it cannot maintain this pressure, contact appropriate instrument support personnel.

b. Ensure that the FID flame is lit and functioning properly.

c. Perform an analysis of the testmix. Open the appropriate testmix instrument method, and verify the parameters as listed in the 'Instrumental Conditions' section of this protocol. Set up a sequence, load the autosampler with a vial containing the testmix, and start the analysis. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the chromatogram.

d. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact appropriate instrument support personnel.

6.2 As Needed Checks

The following steps are to be performed as needed based on system performance. Indicate completion in the appropriate QA/QC log.

a. Replace the septum in the GC injection port.

b. Replace the liner within the GC injection port.

c. Check the GC syringe in the autosampler. Replace if needed.

d. Replace the autosampler bands (if equipped).

e. Perform positive and/or negative CI autotune.

7 Instrumental Conditions

7.1 Gas Chromatograph/Mass Spectrometer

7.1.1 Gas Chromatograph

Oven

Initial Temp:	60°C
Initial Time:	2.0 min
Ramp:	35°C/min
Final Temp:	250°C
Hold Time:	10.0 min
Equilibration Time:	0.5 min

Inlet/Injector

Inj Vol:	1.0 µL
Mode:	Splitless
Inlet Temp:	220°C

Column

Type:	DB-5 MS
Length:	30 m
Diameter:	0.25 mm
Film Thickness:	0.25 µm
Mode:	Constant Flow
Init Flow:	1.2 mL/min
Average Lin Velocity:	40 cm/sec
Carrier Gas:	Helium

7.1.2 Mass Spectrometer

Solvent Delay:	3.0 min
Scan Mode:	Full Scan
Scan Range:	50-500 m/z

Temperatures
Same for EI, PICI, NICI

Transfer Line:	280°C
Source:	200°C – 230 °C
Quad:	150°C

7.2 Gas Chromatograph/Flame Ionization Detector

7.2.1 Gas Chromatograph

Oven

Initial Temp:	100°C
Initial Time:	1.0 min
Ramp:	30°C/min
Final Temp:	260°C
Hold Time:	4.0 min
Equilibration Time:	0.5 min

Inlet/Injector

Inj Vol:	1.0 µL
Mode:	Split
Inlet Temp:	220°C
Split Ratio:	50:1

Column

Type:	DB-5
Length:	15 m
Diameter:	0.25 mm
Film Thickness:	0.25 µm
Mode:	Constant Flow
Init Flow:	1.0 mL/min
Carrier Gas:	Helium

7.2.2 FID

Temperature:	280°C
Mode:	Constant makeup flow
Hydrogen flow:	40.0 mL/min
Air flow:	450.0 mL/min
Makeup flow:	30.0 mL/min
Makeup gas:	Nitrogen

8 Decision Criteria

8.1 Autotune

If an autotune of the mass spectrometer has been performed, verify the results below. Compare the results of the autotune to previous autotune results. Significant voltage increases or changes in the isotope ratios indicate the need to initiate corrective maintenance procedures.

8.1.1 Electron Impact Ion Mode

The following are typical electron impact ion autotune values for the MSD:

a.	PFTBA Tune:	Mass ± 0.4 for m/z 69, 219, and 502
b.	Peak width:	0.45-0.65
c.	Relative abundance:	69 greater than 50% 219 greater than 50% 502 greater than 1%

8.1.2 Positive Ion Chemical Ionization Mode

The following are typical positive ion autotune ($PCICH_4$) values for the MSD:

a.	PFDTD Tune:	Mass ± 0.4 for m/z 41, 267, 599
b.	Peak width:	0.45-0.65

c. Relative abundance: 69 present
267 present
599 present

8.1.3 Negative Ion Chemical Ionization Mode

The following are typical negative ion autotune ($NCICH_4$) values for the MSD:

a. PFDTD Tune: Mass ± 0.4 for m/z 185, 283, 351

b. Peak width: 0.45-0.65

c. Relative abundance: 185 present
283 present
351 present

8.2 Testmix

8.2.1 Gas Chromatograph (regardless of detector type)

Verify the results of the testmix.

a. In order for the instrument to be considered in good operating condition, both Lidocaine and TBEP should generate well-resolved, symmetrical peaks with baseline separation.

b. A SNR of 3:1 will be the minimum response necessary to consider a response a peak.

c. There should be no significant extraneous peaks in the chromatogram.

d. The retention times of each component should be similar as compared to previous analyses (unless GC maintenance has been performed, such as column clipping or replacement).

8.2.2 Mass Spectrometer

In addition to the criteria in section 9.2.1, check the following criteria when using the mass spectrometer:

a. Check for the correct mass assignments for the mass spectra, for EI:

- Lidocaine ions 86 and 234
- TBEP ions 57, 199, and 299

b. Check for the correct mass assignments for the mass spectra, for PICI Source:

- Lidocaine ion 235
- TBEP ions 199, 299, 399

9 Calculations

Not applicable.

10 Measurement Uncertainty

Not applicable.

11 Limitations

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

12 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis. Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

13 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (Inst 001) *Instrument Operation and Systems Support SOP Manual.*

"Gas Chromatograph General Maintenance Protocol" (Inst 002) *Instrument Operation and Systems Support SOP Manual.*

"Mass Spectrometer General Maintenance Protocol" (Inst 004) *Instrument Operation and Systems Support SOP Manual.*

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
0	10/01/12	New document titled "Performance Monitoring Protocol (QA/QC) for the Agilent GC/MS." This document replaces Inst 110 and Inst 306.
1	04/25/16	Section 3 updated for new model information. Section 7.1.2 changed to a range for the source temperature.
2	10/04/18	Updated Section 1 Scope to include applicable disciplines/categories of testing. Updated heading in Section 5. Added 'appropriate instrument support personnel' in Sections 6.1 c, 6.1.1 d, 6.1.2 e, and 6.1.3 a & d. Updated 8.2.1 c & d to account for instrument variation and maintenance. Updated to 'Instrument Operation and Systems Support' in Section 13 and header.

Approval

Redacted - Signatures on File

Drug Chemistry/
General Chemistry
Technical Leader: Date: 09/28/2018

Toxicology
Technical Leader: Date: 09/28/2018

Paints and Polymers
Technical Leader: Date: 09/28/2018

Fire Debris Technical
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